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Magnetic and Low Temperature Electrical Properties of Ni/Fe Multilayers

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Abstract

The magnetic multilayers, [Ni(100nm)/Fe(100nm)]$_n$; n = 1, 2, 3 and 5 were deposited by electron beam evaporation method, under high vacuum at 473 K and investigated for structure, morphology, magnetization and low temperature electrical resistivity from 4.2K to 300K. The structure and grain sizes were determined from the X-ray diffraction (XRD) studies. Microstructures of the films have been examined on the Scanning Electron Microscopy (SEM) images and the surface roughness was determined by atomic force microscope (AFM). The magnetization as a function of field was measured using vibrating sample magnetometer (VSM). The coercivity and remanent magnetization were found to be increasing with increasing number of bilayers and this is ascribed to the magnetic hardening of the films with increasing number of bilayers, n. The resistivity increased with increase of n at all temperatures of interest. The residual resistivity ratio (RRR) and the temperature coefficient of resistivity (TCR) were determined. The power law variation of resistivity with temperature was established. For temperature, *T*, above 80K, the resistivity exponent was found to be slightly more than unity and it was between 3 and 5 for temperature below 30 K. The contributions to resistivity for T above 80K were expected to be largely due to electron-phonon and electron-magnon scatterings and for T below 30K they were expected to be due to electron-electron and electron-defect scattering. This is for the first time that a set of [Fe/Ni] multilayers in the present configurations were probed for structure, magnetic and low temperature electrical resistivity and the power laws for the resistivity variation with temperature have been established.

Keywords

Multilayers; Magnetization; Coercive Field; Resistivity

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Introduction

In the past a decade or so, magnetic and electrical transport studies on magnetic multilayers have been intensely pursued [Hutchings et al. (1999)]. These studies also include investigations on crystal structure, nature of layers and interfaces. For example, [Ni (86Å)/Fe(29Å)]$_{11}$ multilayer was probed for texture and magnetic properties as a function of deposition temperature [Veres et al. (1997)]. The changes occurring in texture, grain size and superlattices with deposition temperature were noted. Nonmonotonic variations in electrical resistivity and coercivity with deposition temperature were also observed. An irradiated multilayer, [Ni(13Å/Fe(33Å)]$_{10}$ with swift heavy ions was studied for magneto-optic Kerr effect and electrical resistivity as a function of temperature [Srivastava et al. (2004)]. The interface mixing leading to alloy phase formation was observed when the films were irradiated. Similar studies were reported for Si^+ ion irradiated Fe(28Å)/ Ni(85Å) multilayers [Rachana Gupta et al. (2004)]. Power laws for the resistivity variation with temperature were established. The multilayers, [Fe(29Å)//Ni(86Å)]$_{10}$ and [Fe(50Å)//Ni(50Å)]$_{10}$ were probed for interfacial properties and magnetization as a function of annealing temperature [Veres et al. (2001)]. The effect of thickness of Fe layer over Ni layer on the magnetization and structural properties were investigated [Sander et al. (1997)]. Experimental results on electrical resistivity and magnetization in Ni-Fe alloy films are reported for the fields applied in parallel and perpendicular to the normal surface [Yeh et al (2008)]. Several multilayer systems of the type [Fe/M]$_n$, M = Cu, Al, Au were studied for magnetic interlayer coupling [Shamsutdinov et al. (2006), Sternik et al. (2007), Brajpuriya et al. (2007), Lhiraoui et al. (2008), Fahmi et al. (2009), Brajpuriya et al. (2008)] and n stands for number of repeats or bilayers. The trilayer systems comprising the layers of magnetic and nonmagnetic metals were intensely explored for nature and variation of coupling between the magnetic

layers [Egelhoff et al. (1992), Aliev et al. (1998)]. The multilayers involving ferro and antiferromagnetic layers of the type, $[Fe/Cr/Fe]_n$ were examined for interfacial roughness, coupling and magnetic properties [Dileep Kumar et al. (2005), Botana et al. (2008)].

The Ni/Fe bilayers are technologically very important because of their soft magnetic and electrical properties such as low coercivity, high squareness and low saturation field. From the above cited literature, it is clear that the comprehensive studies on structure, surface morphology, magnetic and electrical properties of [Ni/Fe] multilayers have not been reported so far. In view of this, we studied structure, magnetic and electrical properties of multilayers, $[Ni/Fe]_n$; n = 1, 2, 3, and 5. The films are labeled as NF1, NF2, NF3 and NF4 respectively.

Experimental

The multilayers, [Ni/Fe] were deposited on to the well cleaned glass substrates at a temperature of 200℃ and a pressure of 5 x 10^{-6} mbar. In the cleaning process, the glass substrates were kept immersed for half an hour in boiling chromic acid solution and then left them for ten hour in the solution. After that they were washed with detergent soap solution and finally rinsed with acetone. Before loading into the vacuum chamber, they were cleaned and dried [Sadashivaiah et al. (2010)]. The Ni and Fe layers were deposited by evaporating the nickel and iron sources from two separate molybdenum crucibles using two independent electron beam guns. The thickness of the layers was measured during deposition with the help of quartz crystal thickness monitor. The films were annealed to room temperature slowly in the vacuum chamber.

Structural investigations were carried out by X- ray diffraction (XRD) studies using Brucker-D8 advance diffractometer with Cu-Kα radiation of 1.5406Å wavelength. The microstructure of the films was probed using Cambridge Instruments Stereoscan (Model 150) Scanning Electron Microscope. Surface morphology of the films was investigated by AFM (Digital Instruments Nanoscope III). Average grain size and surface roughness of the films were determined on a scan area of 1μmx1μm using Nanoscope software. Room temperature magnetization was measured using vibrating sample magnetometer (Model ADE-EV9) with a maximum applied field of 0.1T. The low temperature (4.2K to 300K) resistivity measurements were carried out by following the four probe method using an Oxford Instruments make resistivity setup.

Results and Discussion

X-ray Diffraction (XRD) Studies

The XRD spectra for 2θ between 42° and 48° for NF4 film is shown in Fig. 1(a) and the Gaussian fit to the peak is also depicted in Fig. 1(b). The spectra exhibit single sharp peak and its position does not exactly represent any plane in Fe or Ni crystals and therefore it can be considered to be representing mixed phase of Fe and Ni. No other peaks are visible in the spectrum up to 68^0. The peak positions of these films (Table 1) indicate broadly the (111) texture. The average grain sizes have been estimated using Scherrer's formula [Cullity (1956)],

$$D = \left(0.9\lambda / B\cos\theta_B\right) \quad (1)$$

Where, D is the grain size, B the angular width in terms of 2θ, θ_B the Bragg angle and λ the wavelength of X-rays. The interplanar spacing, d was calculated using the relation,

$$d = \left(\lambda / 2\sin\theta_B\right) \quad (2)$$

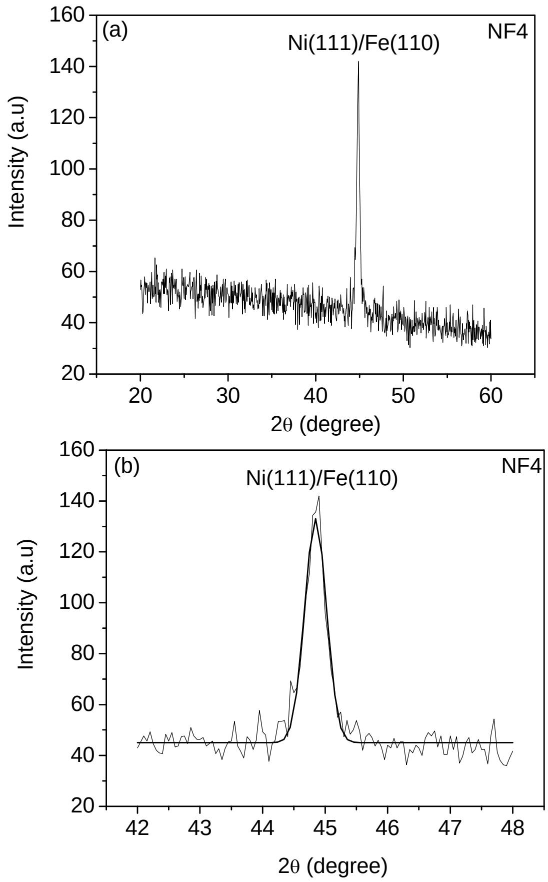

FIG.1. (a) XRD SPECTRA FOR NF4 FILM AND (b) SPECTRA AROUND THE PEAKS POSITION. THE SOLID LINE IN FIG(B) ABOVE IS A GAUSSIAN FIT TO THE PEAK.

The structure of the spectra around the peak for all the films has been analyzed and the grain sizes and inter-planar spacings were determined and recorded in Table 1.

The average grain size increased with increasing number of bilayers (Fig. 2). These observations show that the present films are nanocrystalline in nature. Interplanar distance increased slightly with increase of bilayers.

TABLE 1 THE PARAMETERS EXTRACTED FROM XRD SPECTRA FOR NF FILMS.

Film	Peak position (2θ)	Peak Width (degrees)	Grain size D(nm)	Interpla-nar spacing D (Å)
NF1	44.918^0	0.491	17.50	2.0161
NF2	44.916^0	0.407	21.13	2.0162
NF3	44.907^0	0.378	22.74	2.0167
NF4	44.846^0	0.349	24.60	2.0192

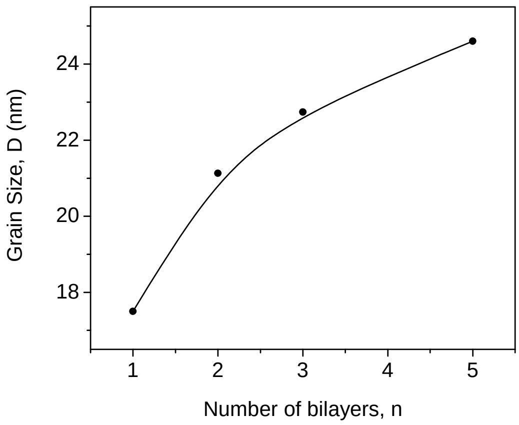

FIG. 2 GRAIN SIZE, D VERSES NUMBER OF BILAYER, N AS OBTAINED FROM XRD. SOLID LINE SHOWN IS A GUIDE TO THE EYE

In reference [Veres et al. (1997)], the grain size of the films deposited at room temperature was 12nm and that for the film deposited at 300℃ was 30nm. The grain sizes of the present films which are grown at 200℃ ranging from 17.50nm to 24.60nm. This implies that the grain sizes of the present films are in the range reported. , Two peaks were observed in the XRD spectrum of a film deposited at temperature 200℃ [Veres et al. (1997)]. No two peaks were observed for any of the present films, which reveals that the texture of the films has not been affected with the number of bilayers, n.

Scanning Electron Microscopy (SEM) Studies

The recorded SEM images of the films shown in Fig. 3 have been used for examining the microstructure of the films. The SEM images appear smooth, compact and fine in structure, which indicates a high content of nanocrystalline particles. The size of the nanocrystallites appears to be increasing with increasing number of bilayers, n.

AFM Studies

The understanding of surface morphology of the present films has been attempted through AFM images which were recorded in contact mode with a scan area of 1μm x 1μm. As an example, the AFM images in 2D and 3D for NF1 film is shown in Fig. 4 (a) and 4(b) respectively. The AFM images have been quantified by recording the height verses distance profiles across the length (white line drawn in Fig. 4(a)) as shown in the Fig. 5 [Aswal et al. (2002)]. This analysis provides information on average grain size (D) (pair of blue dot lines in Fig. 4(a)) and average surface roughness (h) (pair of red and green dot lines in Fig. 4 (a)) of the films. It is observed that D increases with increasing number of bilayers except for NF2 which has larger grains than the remaining three films. The observed trend of increase of grain size with increase of number of bilayers agrees with XRD results. For NF1 and NF2, the grains appears to be spherical in shape and for NF3 and NF4, the grains are cubic like.

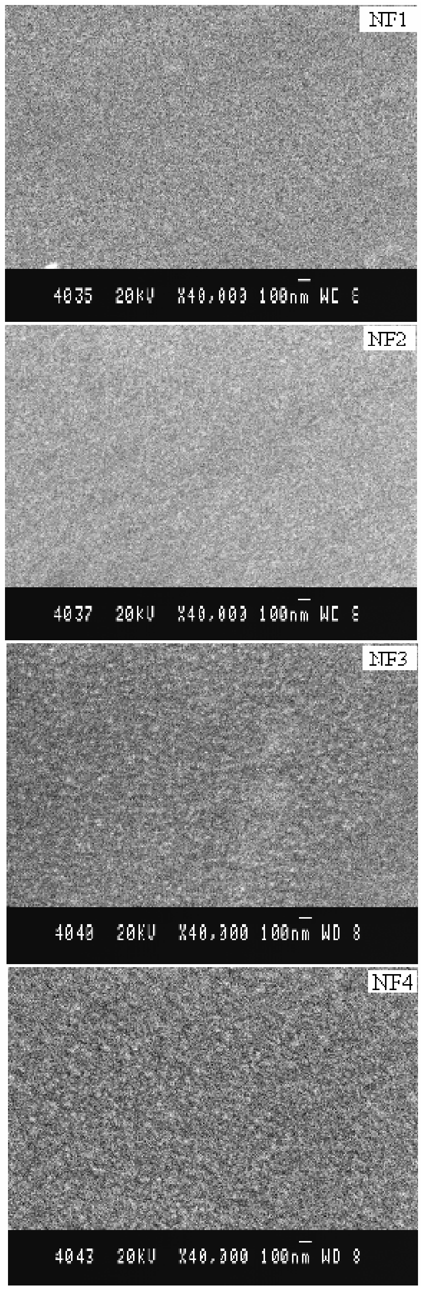

FIG. 3 SEM IMAGES OF THE FILMS

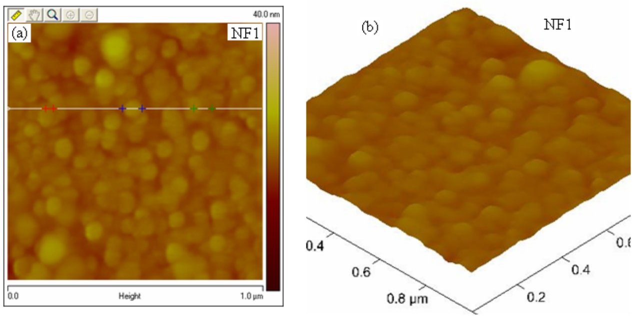

FIG. 4 AFM IMAGES OF NF1 FILMS IN (a) 2D AND (b) 3D.

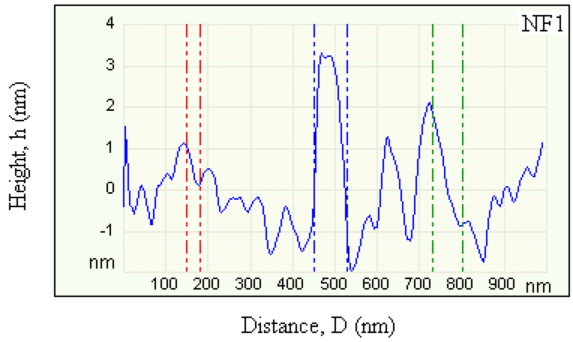

FIG. 5 HEIGHT, H VERSUS DISTANCE, D PROFILE ALONG THE LINE DRAWN IN FIG.4 (a).

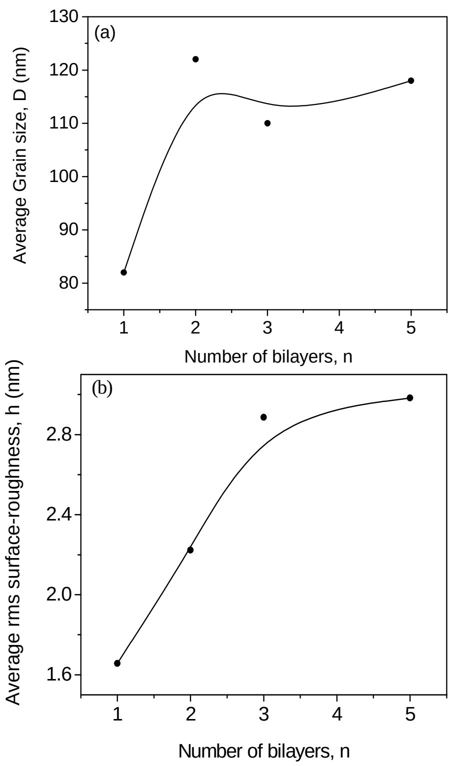

FIG. 6 NUMBER OF BILAYERS, N DEPENDENCE OF (a) AVERAGE GRAIN SIZE, D AND (b) AVERAGE SURFACE ROUGHNESS, H THE SOLID LINES SHOWN ARE THE GUIDES TO THE EYE.

The surface roughness, h, increased with increasing n. The measured D and h values are tabulated in Table 2 and their variations with number of bilayers are shown in Fig. 6. The sectional analysis carried out using the software Nanoscope III, on the AFM images of all the films indicated that grain sizes and their height changes considerably over the distance and this agrees with the results reported in [Sasi et al. (2007)]. Due to this, D and h of the films have been worked out by measuring them for different grains over different cross sections and then averaged.

TABLE 2 AFM PARAMETERS FOR NF FILMS.

Film	Number of Bilayersn	Average grain size, D (nm)	Average rms Surface roughness, h (nm)
NF1	1	82	1.657
NF2	2	122	2.223
NF3	3	110	2.887
NF4	5	118	2.983

It can be noted that D values determined from AFM studies are larger than that of the values obtained from XRD. The disagreement between the grain sizes measured by two different methods could be due to that the measurements on AMF images are based on visual inspection. In case of XRD, it is to do with microscopic scattering of X-rays with lattice points/ planes. This further reveals that the grains observed in AFM images are the agglomerates of many small crystallites that were seen by XRD.

To observe physically such small crystallites in the images, one needs to subject the films to the characterizations like High Resolution Transmission Electron Microscopy (HRTEM) as was done in [Yi et al. (2004)].

Magnetization

The Vibrating Sample Magnetometer (VSM) has been used to study magnetic properties of the films and the effect of number of bilayers on these properties. The room temperature magnetization, M has been measured with field, H applied parallel to the surface of the films. The recorded hysteresis (M versus H) loop for NF1 is shown in Fig. 7. Similar loops were obtained for other films.

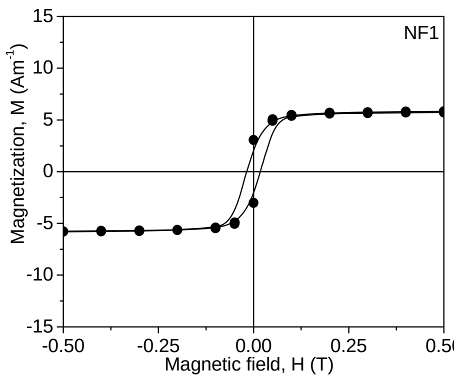

FIG. 7 A PLOT OF MAGNETIZATION, M VERSUS APPLIED FIELD, H FOR ALL FOUR NF1 FILM.

All the four films showed well saturation magnetization behaviour. The typical smoothness of M-H loops indicates highly textured growth of the films. The parameters such as coercive field (H_c), saturation magnetization (M_s), remanent magnetization (M_r), saturation field (H_s), squareness or remanence (M_r/M_s) ratio (s) were determined from the loops and are tabulated in Table 3. The n dependence of these parameters is plotted in Figs. 8 (a-d). The saturation field of 0.1T is commonly observed for all the films which is larger than Ni/Fe films reported in reference [Rachana Gupta et al. (2004)]. It should be noted that the systems investigated by them are different from the present films in terms of thickness of individual layers and n.

TABLE 3. PARAMETERS DERIVED FROM M-H LOOPS OF NF FILMS.

Film	Coercive Field H_c (T) x 10^{-2}	Saturation magneti-zation M_s (Am^{-1})	Remanent Magneti-zation M_r (Am^{-1})	Squareness ratio, s (M_r/M_s)	AF Coup-ling (1-s)
NF1	1.93	5.62	3.05	0.54	0.45
NF2	2.06	8.37	5.35	0.64	0.36
NF3	2.22	7.24	5.52	0.76	0.24
NF4	2.31	11.79	9.39	0.80	0.20

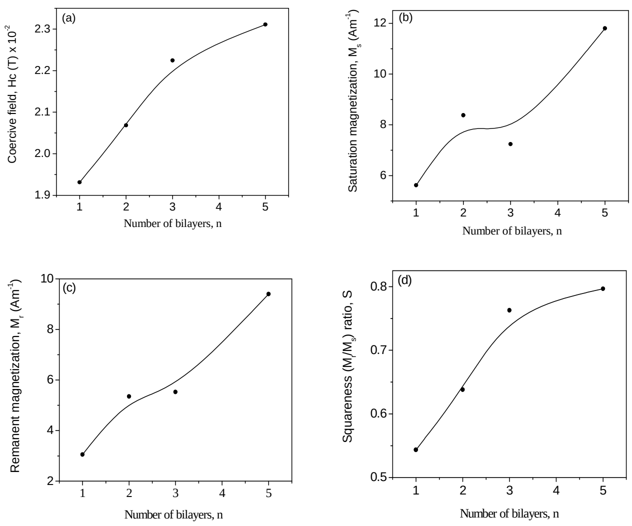

FIG.8. NUMBER OF BILAYERS, N DEPENDENCE OF (a) COERCIVE FIELD, H_C (b) SATURATION MAGNETIZATION, M_S (c) SATURATION FIELD, H_S AND (d) REMANENCE RATIO, S. THE SOLID LINES DRAWN ARE ONLY TO GUIDE THE EYE.

The H_c increased with increasing n. This suggests that the films became more and more magnetically hard with increasing n. The observed increase in H_c can be due to the increase in h and n as measured in AFM studies. The increase of M_s with increasing n can be attributed to the fact that more number of magnetic dipoles are added to the multilayer systems with increasing n and that in turn increases M_s. A close look at Fig. 8(b) it can be noted that NF2 measured slightly more M_s than NF3 and this can be due to grain size effect seen in Fig. 6(a). The remanence ratio, s increased systematically with increasing n, which reveals that there is a structural improvement taking place in the films with increasing n. This is reflected in XRD results as the peak got sharpened with increasing n. Further, the observed (1-s) values for the present films which are the measures of antiferromagnetic (AF) coupling hints at existence of AF type of coupling [Dileep Kumar et al. (2005), Sadashivaiah et al. (2010)] between Fe and Ni layers though both the layers are basically ferromagnetic and there is no spacer layer between them. Here, s =(M_r/M_s). The AF coupling decreases with increasing n as (1-s) decreases with n (Table 3).

Resistivity Behaviour with Temperature

The electrical resistivity, ϱ in the temperature (T) ranges from 4.2 K to 300 K has been measured by following the standard four point method. Due to unavailability of the facility the measurements on the film NF2 could not be made. The measured variation of ϱ with T for the film NF1 is displayed in Fig. 9. Similar nature of variation of ϱ with T has been observed for NF3 and NF4 films. The ϱ increased with increasing n at all temperatures of interest for all the three films. The ϱ of all the three films was found to vary between 0.458 μΩm and 1.027 μΩm for the temperature range from 5K to 300K. The room temperature (300K) ϱ values of the present films were observed to be more by one to two orders of magnitude than the bulk ϱ values of the two components (Ni ~ 0.072 μΩm and Fe ~ 0.09 μΩm). The larger ϱ measured in these films may be due to intermixing of layers at the interface [Ou et al. (2008)] and this was also evident from XRD results.

As per XRD studies, D increases with increasing n. The increase of D also infers that number of grains decreases with increasing n. Due to decrease of grains, the grain boundary scattering reduces with increasing n and consequently ϱ is expected to decrease. Contrary to this, ϱ of the present three films both at 5 K and 300 K (see Table 4) increased with increasing n. In addition to interfacial and grain boundary scatterings, other contributions to ϱ such as electron-impurity, electron-phonon, electron-magnon etc., scatterings can invariably be present. The electron-impurity scattering is usually shows up at low temperatures and electron-phonon and electron-magnon scatterings dominates at high temperatures whereas grain boundary and interfacial scatterings can be present at all the temperatures of

interest.

TABLE 4. THE FITT AND CALCULATED PARAMETERS OF THE NF FILMS.

Film	ϱ(5 K) (μΩm)	ϱ(300 K) (μΩm)	a_1x10^{-10} (μΩ m K^{-k})	k	a_2 x 10^{-7} (μΩ m K^{-m})	m	a_3 x 10^{-4} (μΩ m K^{-n})	n	RRR	TCR x 10^{-3}(K^{-1})
NF1	0.46	0.74	0.16	3.94	1.91	2.71	4.12	1.16	1.60	1.56
NF3	0.48	0.82	0.28	5.03	1.28	2.82	3.56	1.22	1.71	1.73
NF5	0.61	1.03	0.91	4.76	1.51	2.84	5.48	1.18	1.67	1.67

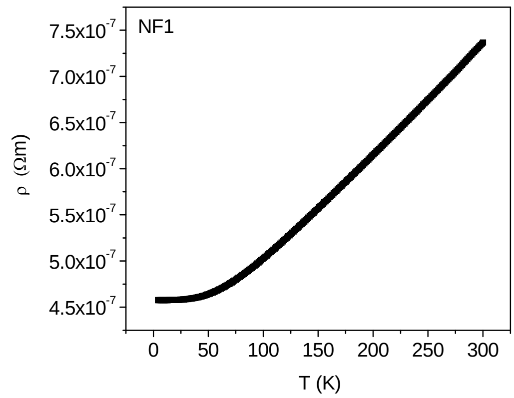

FIG. 9 A PLOT OF RESISTIVITY, ϱ, VERSUS TEMPERATURE, T, FOR NF1 FOR THE COMPLETE TEMPERATURE RANGE

The temperature coefficient of resistivity, TCR = (dϱ/dT)/$ϱ_{RT}$ is determined to be in the range 1.559 x $10^{-3}K^{-1}$ to 1.732 x $10^{-3}K^{-1}$. It may be noted that TCR has been determined for the temperature range from 80K to 300K where ϱ varied linearly with T. The positive TCR of the films points to metallic behavior of the films. The obtained residual resistivity ratio, RRR values of the present three films lie between 1.60 and 1.71 and they are much smaller than that expected for pure bulk metals. The small RRR values observed in these films may be due to enhanced electron-electron, interfacial, grain boundary scatterings etc [Sadashivaiah et al. (2010)].

A close look at the temperature variation of ϱ for the present films revealed that there should exist three different power laws for the measured temperature range. Hence, the following expressions were fit to the data for the different temperature ranges.

$$\rho(T) = \rho(0) + a_1 T^k \quad \text{for T} \le 30\text{K}$$

$$\rho(T) = \rho(0) + a_2 T^m \quad \text{for } 30\text{K} \le \text{T} \le 80\text{K}$$

$$\rho(T) = \rho(0) + a_3 T^n \quad \text{for } 80\text{K} \le \text{T} \le 300\text{K}$$

Where, $\rho(0)$ is the residual resistivity which is taken to be equal to the measured value at 5K in all the films. By regressional analysis, the coefficients, a_1, a_2 and a_3 and exponents, k, m and n were extracted. The fit parameters thus obtained are tabulated in Table 4. The data and the fit curves for NF1 film for the temperature range T ≤ 30K, 30K ≤ T ≤ 80K and 80K ≤ T ≤ 300K are shown in Fig. 10 (a-c). Similarly, the fit curves for NF3 and NF4 films were obtained and are not shown here for saving space.

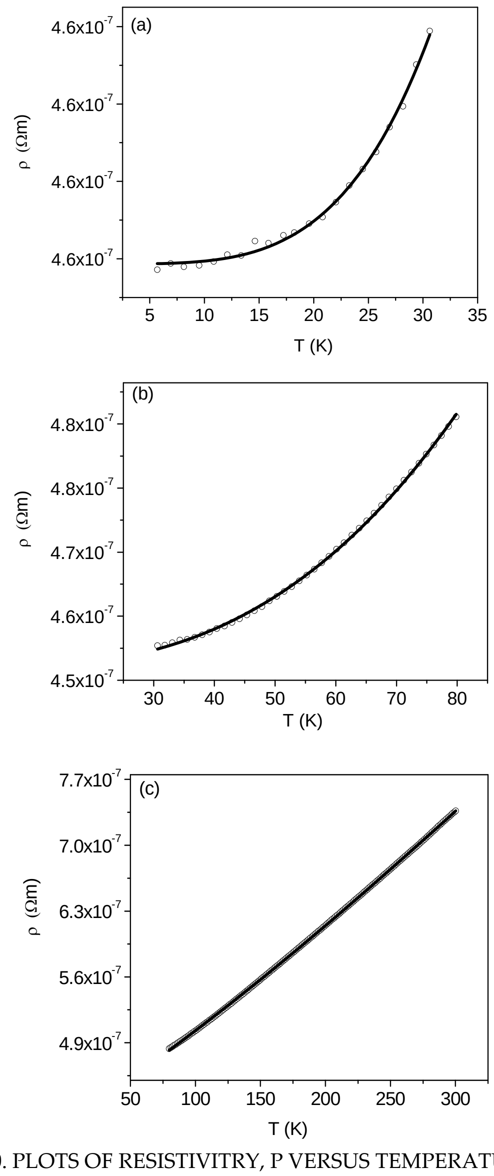

FIG.10. PLOTS OF RESISTIVITRY, P VERSUS TEMPERATURE, T (A). 5K TO 30K (B) 30K TO 80K AND (C) 80K TO 300K. THE CONTINUOUS CURVE PASSING THROUGH THE DATA POINTS ARE FITS TO THE DATA.

For pure nonmagnetic metals, the linearity between resistivity and temperature is expected at high temperature [Taylor et al. (1968)]. For the present films, in the temperature range, 80K≤T≤300K the coefficient, n is obtained to be slightly more than unity, which reveals that in addition to the predominant electron-

phonon scattering there is some contribution to ϱ from electron- magnon scattering also. In Fe/Ni multilayer, the resistivity was found to vary as $T^{1.44}$ for the temperature range from 80K to 300K and this was attributed to additional electron-magnon (s-d) scattering, where s electrons were expected to be scattered by magnons into d band holes [Srivastava et al. (2004)]. For all the three films, in the temperature range, 30K ≤ T ≤ 80K, the exponent, m is found to be between 2 and 3 and that agrees with the results on a magnetic layer [Srivastava et al. (2004)] and deviates from White and Woods value of 3.3 for bulk Fe [White et al. (1959)]. In this range of temperature, electron-phonon s-d scattering dominates over electron-magnon scattering which is expected to be below 20K. For the temperature, T ≤ 30K, the exponent, k is found to be high (in the range from 3 to 5) compared to 2 observed in bulk Fe [White et al. (1959)], Ni [Kondorskii et al. (1958)] and in multilayer [Srivastava et al. (2004)]. This result points out that in this range of temperature, for these films, major contributions are from electron-electron and electron-defects (which includes impurity) scattering along with some contribution from electron-magnon scattering. There are no many reports in the literature on low temperature resistivity of multilayers of present type so that the results on the present films can be directly compared.

Conclusions

i).The Multilayer films, $[Ni(100nm)/Fe(100nm)]_n$; n = 1, 2, 3 and 5 were deposited by electron beam gun method at 473K under high vacuum conditions. The structure, grain size and surface roughness were probed by X-ray diffraction (XRD), scanning electron microscope (SEM) and atomic force microscope (AFM).

ii). At room temperature, magnetization hysteresis loops were recorded in a vibrating sample magnetometer (VSM). The coercivity, H_c and remanent magnetization, M_r increased with increasing number of bilayers indicating magnetic hardening of the films with increasing number of bilayers. The saturation magnetization, M_s increased with increasing bilayers except for NF2 whose M_s was larger than that measured in NF3. This was attributed to the grain size effect.

iii). Electrical resistivity as a function of temperature in the range from 4.2K to 300K has been measured. The residual resistance ratio (RRR) and the temperature coefficient of resistance (TCR) were determined.

iv). Resistivity increased with increasing number of bilayers. This has been attributed to enhanced interfacial scattering with increasing number of bilayers.

v). The power laws for the resistivity variation with temperature were established by subjecting the data for regressional analysis. The power law exponent was slightly more than unity in the high temperature region (80K≤T≤300K) and high in the low temperature region (T ≤ 30 K). It is concluded that in these films, electron-electron and electron-defect scatterings are predominant below 30K and, electron-phonon and electron-magnon scatterings are predominant above 80K.

For the first time that a set of multilayer films of Fe and Ni in the present configurations were investigated for structure, magnetic and electrical properties and the power law variations of resistivity with temperature in the range from 5 K to 300 K have been established.

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